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MODE I INTERLAMINAR FRACTURE TOUGHNESS TESTING OF A CERAMIC MATRIX COMPOSITE (PREPRINT)

G. Ojard and R. Miller

Pratt & Whitney

T. Barnett and M. Dahlen

Southern Research Institute

U. Santhosh and J. Ahmad

Research Applications, Inc.

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MATERIALS AND MANUFACTURING DIRECTORATE
WRIGHT-PATTERSON AIR FORCE BASE, OH 45433-7750
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G. Ojard and R. Miller (Pratt & Whitney)
T. Barnett and M. Dahlen (Southern Research Institute)
U. Santhosh and J. Ahmad (Research Applications, Inc.)

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Pratt & Whitney
400 Main Street
East Hartford, CT 06108

Southern Research Institute
757 Tom Martin Drive
Birmingham, AL 35211

Research Applications, Inc.
San Diego, CA

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Ojard, G.[1], Barnett. T.[2], Dahlen, M.[2], Santhosh, U.[3], Ahmad, J.[3], and Miller, R.[1]

[1]Pratt & Whitney
400 Main Street
East Hartford, CT 06108

[2]Southern Research Institute
757 Tom Martin Drive
Birmingham, AL 35211

[3]Research Applications, Inc.
San Diego, CA

ABSTRACT

As ceramic matrix composites are being targeted for aerospace applications, key material properties need to be understood. This is especially true since the current 2-D architecture-based materials are fabricated by stacking plys that are prime paths for delaminations. Hence, an effort to understand the current test method for delamination toughness was initiated. Specifically, interest was in using a notch for the test and not a starter crack. A model ceramic matrix composite of a SiC fiber with a polymer infiltration pyrolysis derived ceramic matrix was used in this investigation. This material was machined as a double cantilever beam specimen. Testing was done under displacement control. This effort will discuss the testing and analysis of the double cantilever beam specimen.

INTRODUCTION

As interest is expressed in ever increasing higher temperature capable materials, Ceramic Matrix Composites (CMCs) become more attractive than monolithic ceramics since they exhibit the capability to handle damage (increased strain capability) [1]. This is shown by the interest in CMCs for gas turbine engines for turbine and combustor applications [2,3]. Applications are also being pursued in the energy field where improved efficiencies are being pursued such as the use of candle filters [4]. These applications take advantage of the CMC material capability to handle elevated temperatures with limited cooling as well as the improved thermal shock resistance [4, 5]. In the area of propulsion, there is the added benefit of using a lower density material which leads to increased energy efficiencies [5,6].

Even with the potential that CMCs possess, there is a need to fully test and understand the material capabilities. This extends to the delamination capability of the material. Currently, most efforts are supplemented with sub-element testing and increasingly complex rig tests to show that delaminations are not an issue for a given application. Additionally, efforts are undertaken to consider 3D weaves or interlocked architectures to limit delamination growth in ceramic matrix composites [7]. By changing the architecture, the technology readiness level of the material is lowered and additional testing will be required. These approaches can incur significant testing cost. A lot of this testing or consideration of alternate weaves can be reduced or eliminated by determining the material resistance of crack propagation between plies. This

can be done by conducting interlaminar fracture toughness testing of the composite focused on propagating cracks within the matrix of the CMC.

With this in mind, a series of limited tests were undertaken to look into the delamination toughness capability of a ceramic matrix composite [8]. The focus of this effort was to start the testing using a notch as a more reproducible approach and see if the test generated practical results. (The use of a notch and not a starter crack out of a notch would reduce material usage as well.) Additionally, the test results will be compared against results documented by others on other material systems to see if there are some universal lessons learned out of this test method.

PROCEDURE

Material Description

Due to the experimental nature of the testing and the desire to explore variations of 2D weaves and thicknesses, a polymer infiltration pyrolisis (PIP) system was chosen as the model material for this effort. This type of experimental system allows similar fabrication regardless of the weave and thickness. Two different weaves were tested: cross-ply (CP) and quasi. The cross-ply material was either an 8 ply ($[0/90]_{2s}$) or 18 ply layup. The quasi material was either an 8 ply ($[0/90/45/-45]_{s}$) or 16 ply layup. The room temperature average properties for these two layups are shown in Table I. The test results indicate that the in-plane elastic modulus is relatively independent of ply lay-up (cross-ply or quasi).

Table I. Room Temperature Tensile Properties (In-Plane Properties)

Lay-Up	Modulus (E11)	Ultimate Tensile Strength	Strain-to-failure
	GPa (StDev)	MPa (StDev)	mm/mm (StDev)
Cross Ply	111 (7)	246 (30)	0.004 (0.001)
Quasi	107 (12)	199 (16)	0.0036 (0.001)

Specimen

To machine the double cantilever beam specimens, the specimens were initially machined into rectangles that were 101.6 mm in length and were 12.7 mm in width. Specimen size was based on availability of material. The specimen thickness was not modified during the machining and was left in the as-received state. To fabricate the notch, diamond sawing was done to a depth of 25.4 mm. This is shown schematically in Figure 1. An actual machined notch is shown in Figure 2. The notch was centered in the thickness and the average thickness of the notch was 0.66 mm.

Testing

The schematic for this test is shown in Figure 3. This testing configuration is taken from ASTM D5528 [8]. Loading was done using loading blocks which where adhesively attached to the specimen. The piano hinge approach was not used. The crack opening displacement was measured using extensometer arms as shown in Figure 4. All testing was done under displacement control and the data was recorded electronically.

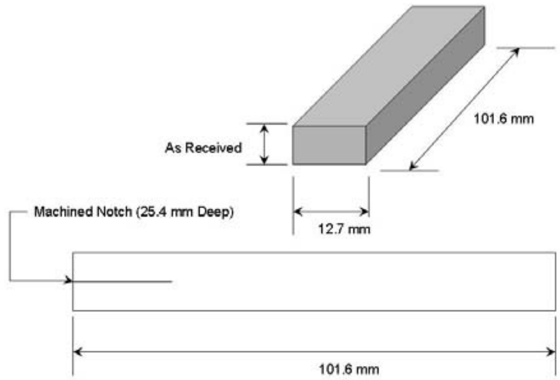


Figure 1. Specimen schematic

Figure 2. Macro image of specimen focused on centered notch

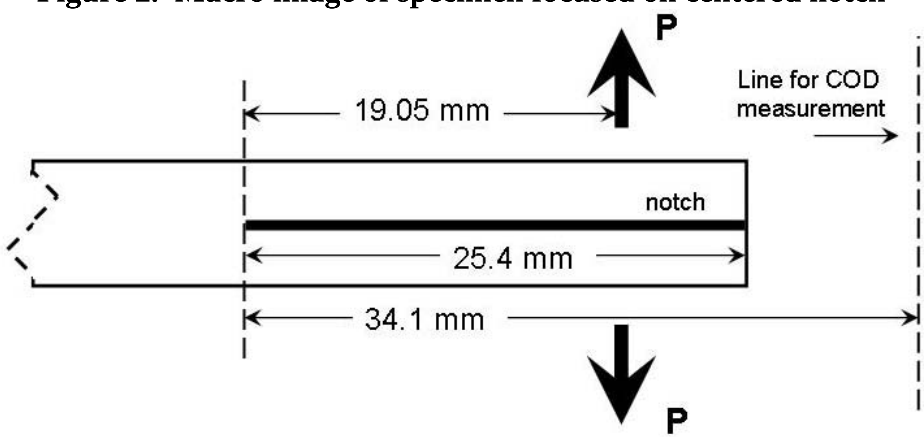


Figure 3. Test schematic showing loading scheme
(loading done with loading blocks)

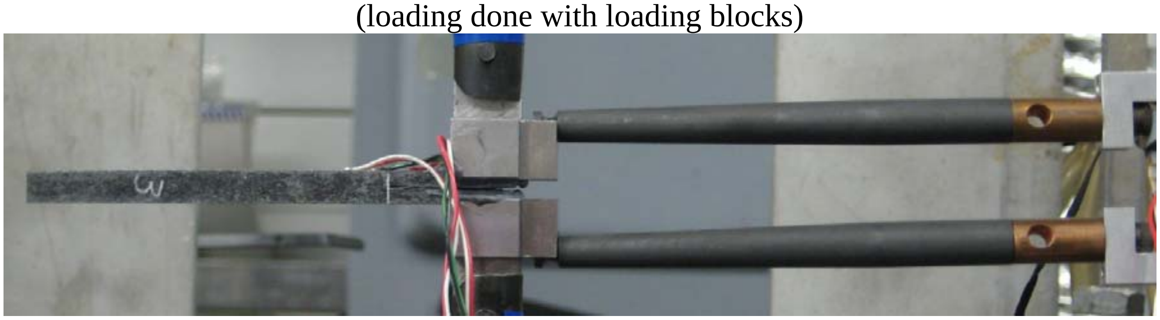


Figure 4. Macrograph showing measurement approach for crack opening displacement
(extensometer arms are held within a groove machined into the loading blocks)

During the testing, detailed images were taken periodically during the loading process on one side of the specimen to document the progress of cracks emanating from the machined notch. These photos were carefully reviewed to allow determination of the crack length generated. This would allow generation of the strain energy release rate (G) for different crack lengths.

The data generated from the testing effort was calculated per the procedures discussed in ASTM D-5528 [8]. The data analysis presented in this paper is from the Modified Beam Theory (MBT) Method [8]. The other approaches for determining the Interlaminar Energy Release Rate (G_I) will not be presented.

RESULTS

8 Ply Material Specimens

The cross-ply and quasi material specimens that were 8 plies thick had an average thickness of 3 mm. With the notch width of 0.66 mm, the resulting arms for the specimen had a thickness of 1.2 mm. This made the arms very slender and during setup for testing it was clearly noted that slight loads resulted in large crack opening displacements. This can clearly be seen in Figure 5 (specimen photo under load) and Figure 6 (load versus crack opening) for the different 8 ply material tested (cross-ply and quasi). Data analysis proved difficult as the data was not consistent with other data sets (to be discussed next for thicker specimens) and it was clear that large displacement corrections were required (per ASTM procedure [8]). These corrections for both lay-ups were large and were multiplicative. It was clear that the 8 ply data could not be confidently presented as the corrections applied did not bring the data into the data range seen for the higher ply count specimens. No additional analysis was done on the 8 ply tests performed but future numerical analysis may be able to correct the data (for cross ply and quasi layups).

16 and 18 Ply Material Specimens

As noted previously, the quasi material was also made as a 16 ply layup with an average thickness of 5.96 mm while the cross-ply material was made as an 18 ply layup with an average thickness of 6.86 mm. With the notch width of 0.66 mm, this meant that the average arms for the DCB test were 2.6 mm in thickness or greater (double the 1.2 mm for the 8 ply material). This increased thickness generated less deflection compared to the 8 ply material as shown by the crack opening displacement (COD) data as shown in Figure 7 as well as a representative image form testing as shown in Figure 8. (COD shown in Figure 7 is half that shown in Figure 6.)

Figures 7 and 8 clearly show that there is not as much deflection as reported for the 8 ply material. The crack opening displacement for the thicker material is half that of the 8 ply material. Therefore, there was no need for corrections to the collected data.

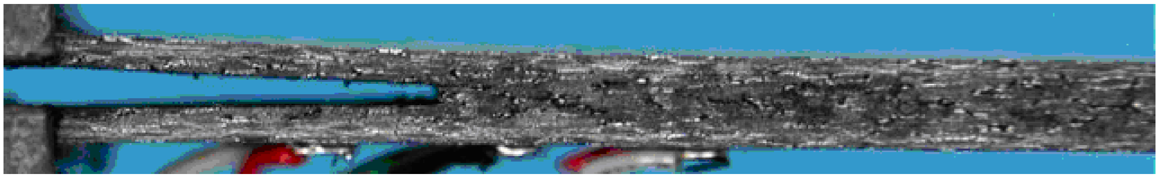


Figure 5. Image from 8 ply quasi specimen in Figure 6 showing large deflection
(Overall (and initial) specimen thickness is 1.2 mm)

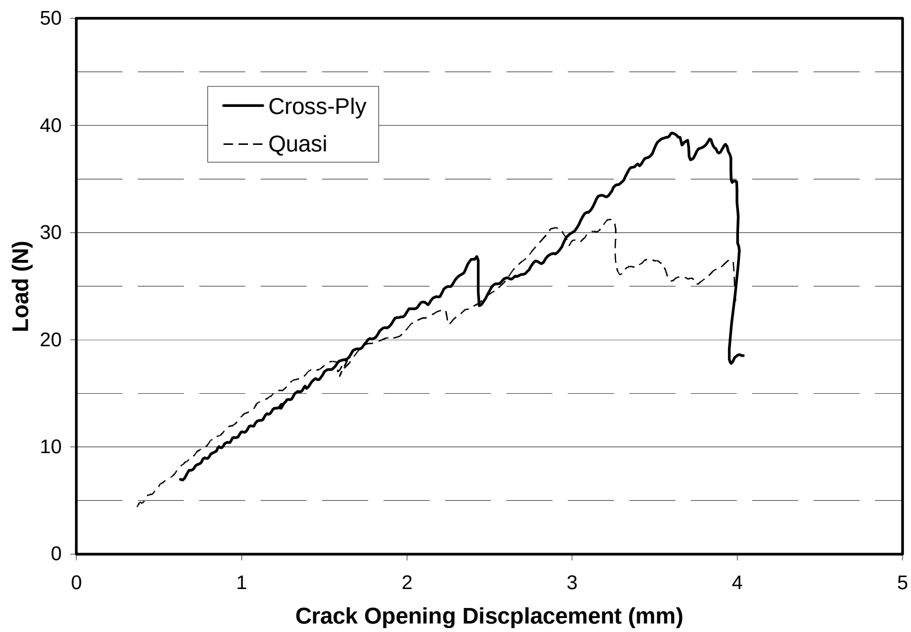


Figure 6. Load versus crack opening displacement for an 8 ply quasi specimen
(raw data shown – not corrected for offset of extensometer arms)

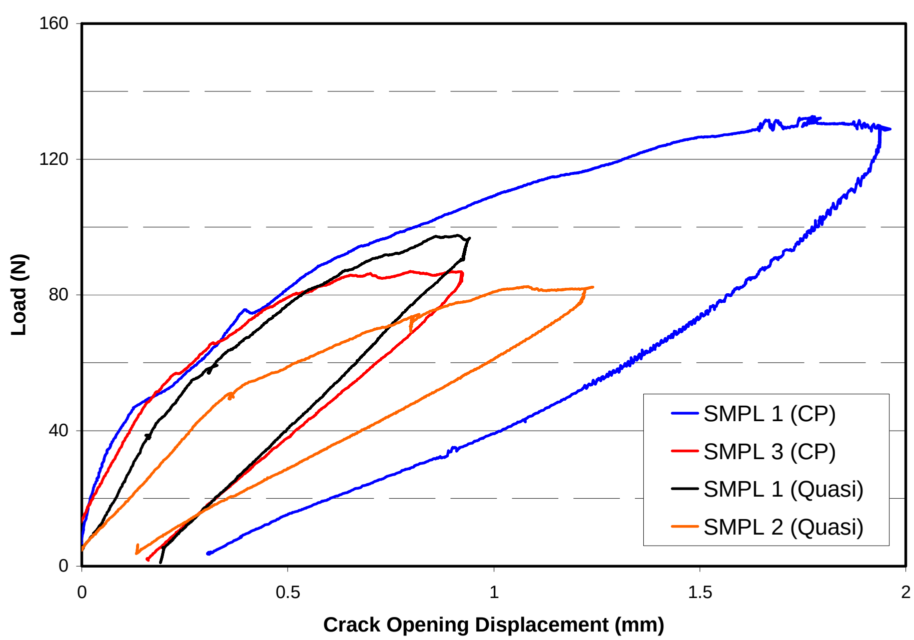


Figure 7. Load versus crack opening displacement for 16 and 18 ply specimens
(Quasi at 16 ply and Cross-ply at 18 ply (raw data shown))

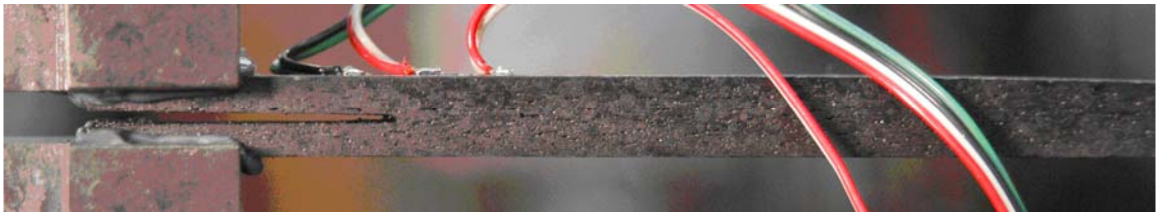


Figure 8. Test image during testing from 18 ply specimen
(specimen thickness of 6.86 mm)

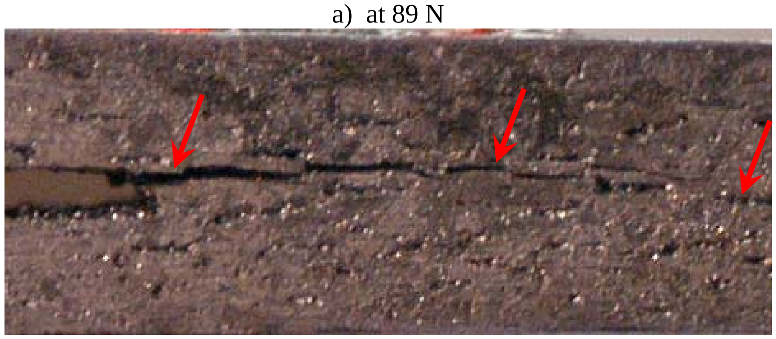
During the testing, photos were taken at increasing load levels to monitor crack formation and length (See Figure 9). Figure 9 shows that the porosity required careful review of the specimens to be sure that the crack was being tracked. The results of this for the specimens are shown in Table II. This allowed the determination of the Interlaminar Energy Release Rate (G_I) for various delamination lengths. Here, the crack opening displacement was corrected. As shown in Figures 3 and 4, the COD was measured offset from the load line and by using the method of similar triangles; the COD was corrected to the value shown in Table II. (The data is not corrected for the graphical images such as Figures 6 and 7.)

In reviewing the calculation approaches listed in the ASTM standard [8] and considering the slight difference between the methods discussed, it was decided to use the correction to the Modified Beam Theory. For this analysis, the Interlaminar Energy Release Rate (G_I) is given by:

$$G_I = 3P\delta / 2b(a + |\Delta|)$$

where P is the load, δ is the load point deflection (or opening displacement), b is the specimen width, a is the delamination length and Δ is a delamination length correction. This correction is determined experimentally by generating a least squares plot of the cube root of the compliance versus delamination length [8]. (The compliance is determined by the ratio of the load point displacement to the applied load (δ/P).) The delamination behaves as if it is a longer delamination by the Δ length. (Δ is determined by finding x-intercept of the least squares fit line.) Figure 10 shows that ~8.8 mm needs to be added to the delamination length for the G_I calculation shown above (previous equation).

a) at 89 N



b) at 129 N

Figure 9. Images from SMPL 1 (CP) at different load levels

Table II. Measurements Taken of Specimens during Testing

Specimen Identification	**Load** (N)	**Crack Length*** (mm)	**COD**** (mm)
CP SMPL 1	89	22	0.35
CP SMPL 1	111	26	0.66
CP SMPL 1	129	33	1.13
CP SMPL 2	49	20	0.11
CP SMPL 2	80	22	0.26
CP SMPL 2	102	25	0.46
CP SMPL 3	71	20	0.23
CP SMPL 3	80	22	0.30
CP SMPL 3	87	27	0.45
CP SMPL 4	49	20	0.14
CP SMPL 4	62	22	0.29
CP SMPL 4	71	27	0.48
Q SMPL 1	58	20	0.17
Q SMPL 1	93	27	0.51
Q SMPL 2	73	22	0.47
Q SMPL 2	82	26	0.68

* = machined notch length and subsequent crack emanating from the notch (from the load line)
** = COD is corrected for extensometer arm being offset from load line

Additionally, this approach allows a check on the approach by determining the modulus of the material [8]. The modulus can be determined by:

$$E = 64((a + |\Delta|)^3 P / \delta b h^3$$

where h is the specimen thickness. Consistent with this approach, the modulus was found to be independent of the delamination length [8]. For this effort, the modulus for the material was determined to be 130 GPa (+/- 30 GPa). This value is higher than the data shown in Table I but with the wide scatter seen, it is within the range reported earlier in Table I. (Table I is also based on a larger data set and 130 GPa is on the high end of the data but outside the standard deviation.)

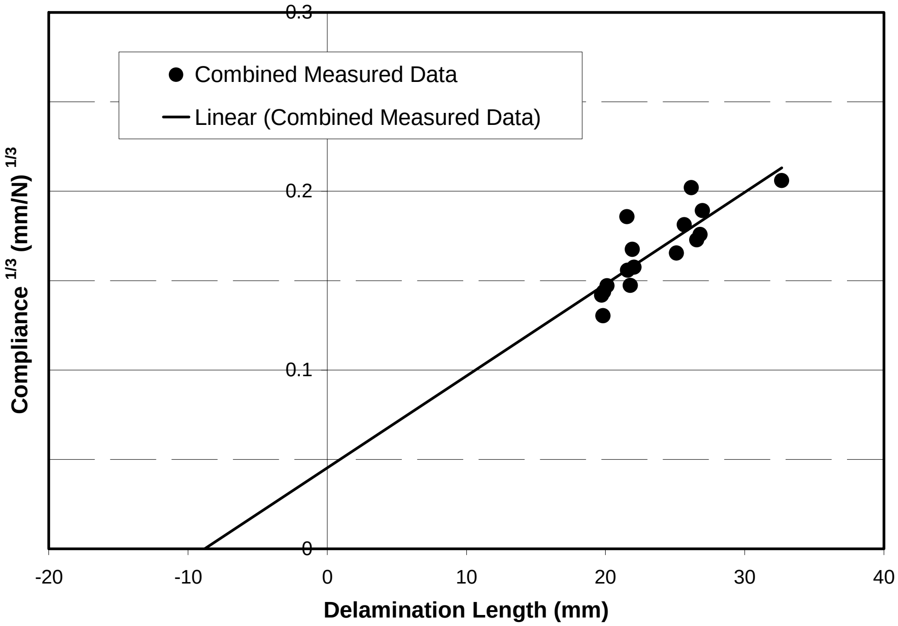


Figure 10. Determination of Delta (Δ) for the Specimens Tested

The resultant interlaminar energy release rate versus crack length was determined for all the specimens tested for varying crack lengths. The resultant data is shown in Figure 11. As the crack length increases, there is a greater range of scatter seen in the data.

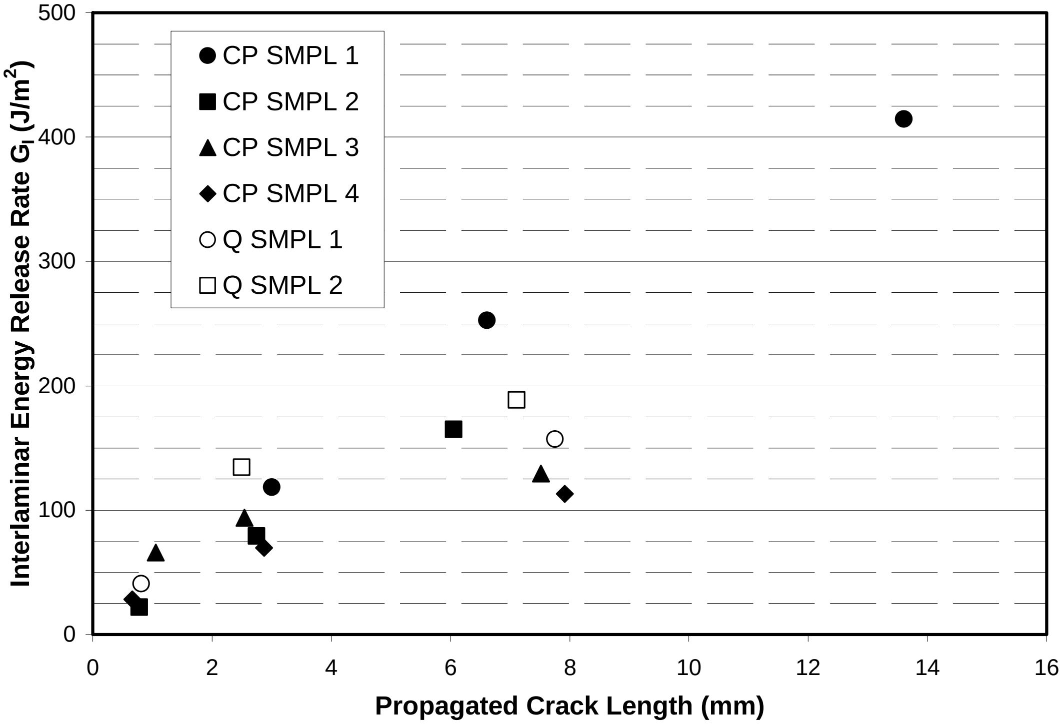


Figure 11. Interlaminar Energy Release Rate versus crack length for the specimens tested (16 ply quasi material and 18 ply cross-ply material)

There was an outlier in the data that was not included in the analysis shown in Figure 11. One cross-ply specimen (SMPL-CP-5) had a significant crack propagate at a low load. This is shown in Figure 12. The load to propagate this crack was 39 N and this is low compared to the loads shown in Figure 7.

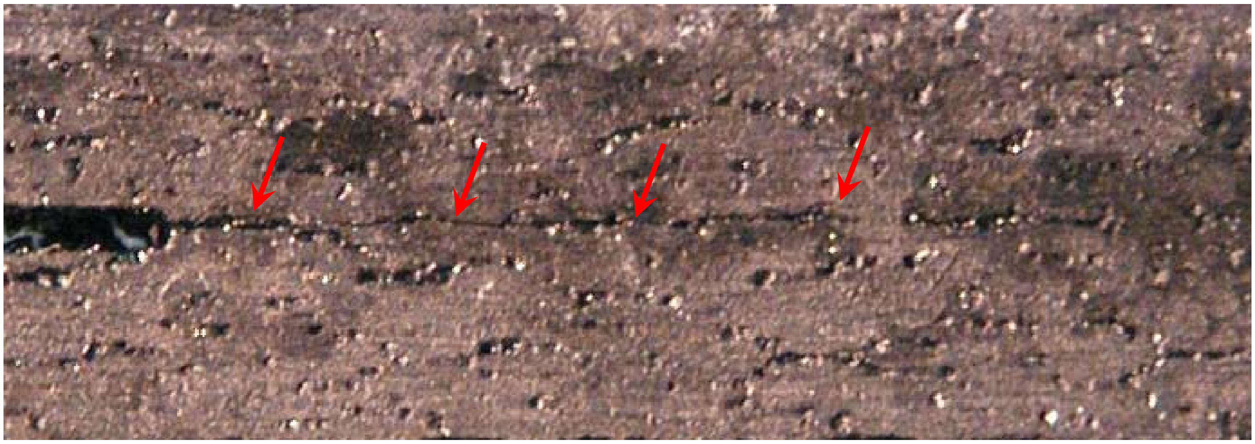


Figure 12. Image from SMPL 5 (CP) at 39 N

All of the above work looked at the Interlaminar Energy Release Rate. What is of greater interest is the Interlaminar Fracture Toughness (G_{IC}). To generate this, the load versus crack opening displacement were linearly fit from a load of 20-35 N and then that line was extrapolated along the curve and where the load deviated from the linear line was recorded and

then used to generate the G_{IC} values. This data was analyzed using the standard Modified Beam Theory (there was no delta applied to the delamination (crack) length). The equation for the Modified Beam theory is as follows:

$$G_I = 3P\delta / 2ba.$$

The results of this testing are shown in Table III. The data for the two layups overlaps, even for this limited number of quasi specimens. Therefore, differences in the Interlaminar Fracture Toughness (G_{IC}) are inconclusive between layups.

Table III. G_{IC} Values –Modified Beam Theory (Standard)

Specimen	Layup	G_{IC}	Average	St Dev.
		(J/m^2)	(J/m^2)	(J/m^2)
SMPL-CP-1	Cross Ply	7	18	8
SMPL-CP-2	Cross Ply	18		
SMPL-CP-3	Cross Ply	24		
SMPL-CP-4	Cross Ply	23		
SMPL-Q-1	Quasi	21	38	24
SMPL-Q-2	Quasi	55		

DISCUSSION

8 Ply Material

The testing of the 8 ply material showed very large deflections, requiring significant data corrections. In similar testing, the use of such corrections by other investigators was not reported [9,10]. This is a combination of two factors: (1) the use of cut notches versus pre-generated cracks made the arms of the double cantilever beam relatively thin; and (2) the lower modulus of the material system used in this study versus the modulus values reported by other investigators [9]. This may indicate a lower limit of modulus when testing thin (e.g., 8-ply) specimens. The testing comparison here indicated that for thin material, sharp pre-cracks are needed in order to keep the arms of the double cantilever beam as thick as possible. Such limitations to a double-cantilever test raise concerns for test reliability. If pre-generation of cracks are required for thinner specimens, for example, this could lead to highly variable testing conditions and the corresponding need for more material. As noted above for the outlying specimen, a crack jump of 20 mm or greater can occur, leading to ineffectual consumption of material when performing characterization efforts on high cost material.

16 and 18 Ply Material

The testing on the thick specimens generated G_I (Interlaminar Energy Release Rate) values from 20 to 420 J/m^2 (See Figure 12). These values are within he range documented by other investigators but on the low end for the multiple SiC/SiC CMC systems tested where G_I was

found to range from 200 to 600 J/m^2[9]. The work reported by others was on CMC systems that were either fabricated by Chemical Vapor Infiltration (CVI), Hot Press (HP) or Melt Infiltrated (MI). These systems all create matrices that do not have the remnant PIP cracks present from repeated infiltrations cycles. While porosity may be a concern in the comparison here, it should be noted that CVI systems may have a matrix that has double the porosity of a PIP system

Interlaminar Fracture Toughness – G_{IC}

The initiation values were determined for the series of tests run. The results are reported in Table III. For this analysis, the standard Modified Beam Theory was used based on the limited damage and crack rotation expected at the start of the test. The values reported in Table III are low but they are consistent with an extrapolation of the data shown in Figure 12 when looking at a zero propagated crack length. (The value is also consistent with the MBT analysis of the large crack extension seen in the outlying specimen discussed above (See Figure 12).)

Additionally, some early testing data generated by the authors was reviewed to determine the interlaminar fracture toughness to see how it compared against the data shown in Table III. At the start of testing, a series of tests were run without documenting the crack growth during the test. These tests were run as a check of the testing approach since this material had an accidental intermediate temperature oxidation exposure done to it that showed debited properties from expectation in standard tensile testing. Both 18 ply cross ply and 16 ply quasi material was tested. The G_{IC} values from the cross ply testing was determined to be 28 and 34 J/m^2 (there were two tests) while the G_{IC} value from the quasi testing was determined to be 42 J/m^2 (there was only one test). These values are in line with the results reported previously in Table III. This indicates that the embrittelment seen from the intermediate temperature oxidation was limited to the interface coating and did not affect the matrix of the material. Additionally, the shape of the curve seen during testing was consistent with the unaffected material that was presented in Figure 7.

CONCLUSION

A series of double cantilever beam tests were done on a Polymer Infiltration Pyrolisis system with varying thickness and ply count. It was clear that a machined notch, engineered for test repeatability, did not yield acceptable crack propagation in 8 ply test specimens. Testing of thicker specimens, of varying crack length and specimen layup, had G_I values from 20 to 420 J/m^2. The range of values arrived at for the model material system are on the lower end of documented results for a range of Ceramic Matrix Composites fabricated by chemical vapor infiltration or melt infiltration means [9]. This is due to the heavily cracked nature of a Polymer Infiltration Pyrolisis fabricated CMC with the repeated thermal cycles that the material is subjected to during manufacturing. This is also shown in the average G_{IC} value of 45 J/m^2that was determined (Average of all values reported in Table III).

This effort did show that a notched approach could be considered for such testing but not for thin materials (as noted above). The appropriate thickness will vary from CMC to CMC based on material properties and manufacturing methods. For the series of tests conducted here, the corrected Modified Beam Theory approach was used. Additionally, the method presented a

way to check the approach by allowing the modulus of the material to be calculated. This is a clear benefit on such a challenging test.

FUTURE WORK

Additional data collected during the testing has not been analyzed at this time. The strain gauges placed on the specimen are still available for data analysis and will be considered for future work. Additionally, crack propagation and micromechanical modeling and analysis is planned and will be presented in the future. A numerical approach may be pursued to help determine the deviation from linearity that was used in the G_{IC} evaluation.

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REFERENCES

1. Brewer, D., 1999, “HSR/EPM Combustion Materials Development Program”, Materials Science & Engineering, 261(1-2), pp. 284-291.
2. Brewer, D., Ojard, G. and Gibler, M., “Ceramic Matrix Composite Combustor Liner Rig Test:, ASME Turbo Expo 2000, Munich, Germany, May 8-11, 2000, ASME Paper 2000-GT-670
3. Verrilli, M. and Ojard, G., “Evaluation of Post-Exposure Properties of SiC/SiC Combustor Liners testing in the RQL Sector Rig”, Ceramic Engineering and Science Proceedings, Volume 23, Issue 3, 2002, p. 551-562.
4. K.K. Chawla (1998), ***Composite Materials: Science and Engineering, 2nd Ed***., Springer, New York
5. Bouillon, E.P., Lamouroux, F., Baroumes, L., Cavalier, J.C., Spriet, P.C. and Habarou, G., 2002, “An Improved Long Life Duration CMC for Jet Aircraft Engine Applications”, ASME paper No. GT-2002-30625.
6. M.F. Ashby and D.R.H. Jones (2001), **Engineering Materials 1: An Introduction to their Properties and Applications, 2nd Ed**., Butterworth Heinemann, Oxford
7. Yun, H. M., DiCarlo, J. A., and Fox, D. S., “Issues on Fabrication and Evaluation of SiC/SiC Tubes with Various Fiber Architectures”, High Temperature Ceramic Matrix Composites 5, pp. 537-542. 2005
8. ASTM D-5528-01, “Standard Test Method for Mode I Interlaminar Fracture Toughness of Unidirectional Fiber Reinforced Polymer Matrix Composites”, ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA.
9. Choi, S.R., and Kowalik, R.W., “Interlaminar Crack Growth Resistances of Various Ceramic Matrix Composites in Mode I and Mode II Loading”, Journal of Engineering for Gas Turbine Power, May 2008, Vol. 130
10. Hojo, M., Yamao, T., Tanaka, M., Ochiai, S., Iwashita, N., and Sawada, Y., “Effect of Interface Control on Mode I Interlaminar Fracture Toughness of Woven C/C Composite Laminated”, JSME International Journal, Series A, Vol. 44, No. 4, pp. 573-581, 2001